

Monsanto

APR 19 1982



FROM : R. T. Parker - Nitro Plant - #507
NAME-LOCATION-PHONE:

DATE : April 13, 1982

cc. H. S. Scott - E2ND
T. M. Bistline - E2NJ
H. M. Galloway

SUBJECT : TCDD ANALYSES

REFERENCE :

TO : A. M. Ford
A2SA

ATTORNEY WORK PRODUCT
ATTORNEY-CLIENT PRIVILEGE

Regarding your inquiry about the TCDD levels reported in the 1969 Nitro TSD Annual Summary, we have no documents that can verify or determine the source of these numbers. The TSD summary reports that TCDD levels averaged 40-50 ppm during 1968. The Nitro research notebooks of Phil Balderson and Marty Wilson contain no analytical data on TCDD levels during this time period. Balderson stated that if any TCDD analyses were run at Nitro, the results would be recorded either in his notebook or Marty Wilson's notebook. Balderson and Wilson were apparently the only Nitro chemists to run analyses for TCDD. Rick Atkinson, the only other Nitro chemist working with 2,4,5-T during 1968, stated he does not recall having run an analysis for TCDD in 2,4,5-T. These facts suggest that no TCDD determinations were made at Nitro during 1968.

Nitro measurements of TCDD levels in 2,4,5-T were carried out in both the years before and after 1968. These measurements suggest that the 40 to 50 ppm figures cited in the TSD summary are not realistic (assuming the Nitro analytical method is reasonably accurate). Nitro measurements never exceeded 28 ppm, and TCDD values typically averaged 12 ppm or less. The attached notebook pages comprise all of the Nitro analytical data I could find on TCDD analyses. I have included some comments on these copies in parenthesis. In particular, I suspect the numbers reported in Marty Wilson's notebook may be questionable. The numbers are quite low and certain samples run by both St. Louis and Marty do not agree. The St. Louis numbers cited in Marty's notebook were probably generated by Bill Udell (currently with Monsanto Agricultural Division). It should be noted that Phil Balderson employed a slightly different method from Marty Wilson for TCDD analyses, and Balderson's method was based on a method obtained from Dow.

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C32902

Another possible source of TCDD analytical data would be Bill Udell. Apparently, he ran TCDD analyses for Corporate Research around the time period in question. The TSD summary might be based on numbers generated by Udell. It should be noted that lots of 2,4,5-T produced in 1968 should begin with the code letters "NK" and this can be used to determine if analytical results correspond to material made in 1968.

Finally, the report in the 1969 TSD summary was probably generated by R. L. Dillon, Jr. and/or J. C. Strum. These engineers were handling process changes, etc. at that time. I questioned Dillon (currently superintendent of MHA operations at Nitro) and he said he did not recall the source of the numbers. Strum is no longer working for Monsanto. It is quite possible the numbers they reported are erroneous.

Terry Parker
R. T. Parker

sa

Attachment

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C22903

98

7/18/66

DIOXANE IN T

PRODUCTION REQUESTED THAT WE
 RUN SAMPLES OF FINISHED T
 FROM DECEMBER '65 THROUGH
 PRESENT PRODUCTION TO DETERMINE
 IF THIS MATERIAL IS BUILDING UP.
 RUN ON FM 720 @ 250°C,
 INJECTOR 330°C, BLOCK 320°C, 3 METER
 3% QF-1 ON AW+HMDS CHROMOSORB G

 DIOXANE @ 2.6 min RETENTION

WEIGH OUT 10 GRAMS 2,4,5-T
 ADD 10 ml CHCl_3 + SHAKE 10
 MINUTES. FILTER AND WASH THE FILTER
 CAKE WITH 5 ml. CHCl_3 . TRANSFER
 FILTRATE TO A VIAL AND ADD 5 ml/
 2 M HNO_3 . SHAKE 10 MIN. DILUTE
 TO 25 ml WITH WATER AND
 CENTRIFUGE. SEPARATE OFF THE ORGANIC
 LAYER AND EVAPORATE DOWN TO 2 ml.
 INJECT 40 μ l INTO THE INSTRUMENT
 @ AN ATTENUATION OF X1.

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4

022904

Note: This is the first Nitro attempt
 to run TCOD analyses per

Balderson.

PSS 4/12/82

NH 12 - 026	12 ppm
NH 01 - 024	6 ppm
NH 02 - 036	10 ppm
NH 03 - 054	7.5 ppm
NH 04 - 004	8 ppm
NH 04 - 028	3.5 ppm
NH 05 - 029	12 ppm
NH 05 - 033	11 ppm
NH 06 - 020	13 ppm
NH 06 - 067	17 ppm
NH 07 - 016	21 ppm
NH 07 - 038	28 ppm
NH 07 - 052	21 ppm

NH 08 - 016

5 ppm

8/12/66

NH 08 - 024

11 ppm

8/12/66

2/2/66
7/12/66

7/21/66
P.23

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C22905

1/23/67

T.O.D. IN 245-T

PROCEDURE SAME AS ON
PAGES 78-79.

BATCH	P.P.M. T.O.D.
NH09-011	3
NH09-051	4
NH10-022	12.5
NH10-082	18
NH11-010	5
NH11-060	3
NH12-009	5
NH12-051	7
NI01-004	4
NI01-021	3

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C32906

TOO IN T ANALYSES

2/17/67

PROCEDURE SAME AS ON
PAGES 48-79. CALCULATION.

$$\frac{\text{PEAK HEIGHT SAMPLE} \times 15 \times 2 \times 1000}{\text{PEAK HT. ST.}} = \text{ppm}$$

NH09-085	8 ppm
NH09-100	12
NI02-001	nil
NI02-010	nil
NI02-020	TABLE
NI02-030	9
NI02-040	1
TCP. B#4 2/16/67	5

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022907

TLP - T - TOD ANALYSES

NEED METHODS TO RUN ALL THESE MATERIALS ON ONE INSTRUMENT.

QF-1 ON CHROMOSORB G WORKS WELL WITH PROGRAMMING FOR 3,4,5-T ALID. THE SAME COLUMN WORKS WELL FOR TOD @ 250° 130. TLP HOWEVER TAILS AND AS THE COLUMN AGES IT TAILS BADLY.

SILYLATION OF THE TLP WAS TRIED BUT ANISOLE IS ONLY SLIGHTLY SEPARATED FROM THE TLP.

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C22908

21

(Balderson NB #7)

4/3/67

TOD IN 245-T

41

10 GRAM SAMPLE ADD 10
ml CHCl_3 , SHAKE 15 min. FILTER
+ WASH 5 ml CHCl_3 . ADD 5 ml
2 M NaOH TO FILTRATE + SHAKE
10 min. CENTRIFUGE WITHDRAW
BOTTOM LAYER. EVAPORATE DOWN
TO 2 ml. RUN ON 2 METER
GF-1 @ 250°C + 120 ml/min
He. 40 μ l ON WX FILAMENTS

LOT	TOD
03 -001	1 ppm
010	3
020	14
030	1
040	7
045	8
050	15
060	15

PK. HEIGHT SAMPLE X 15 X 2 ~~1000~~

PK. HT. STD

= ppm TUU

022909

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4

(Balderson NG #7)

4/17/67

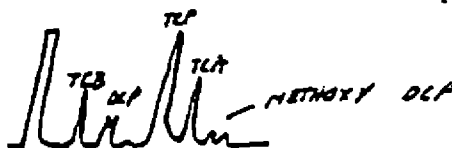
2,4,5-T - ANALYTICAL METHODS

ATTEMPTING TO FIND V.P.C
COLUMNS SO AS TO RUN 2,4,5-T
ACID, TLP & TDD ON ONE
INSTRUMENT.

FFAP ON AW-DMCS
CHROMOSORB 6 TRIED APPEARED
TO WORK WELL AT FIRST BUT
AFTER SEVERAL SAMPLES COULD
NO LONGER OBTAIN T-ESTER
PEAK. HMDS HELPED SOMEWHAT
FOR ONE SAMPLE BUT DID
NOT LAST.

4/20/67

3% SE-54 ON AW-DMCS CHROMOSORB
6. WORKS OR SEPARATES WELL BUT
THE PHENOLS TAIL TOO BADLY FOR
A GOOD ANALYSIS.



PROGRAMMED 100° → 200° @ 7.5°/min

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622910

T
THEN TRIED ADDING B.S.A. TO ACIDIFIED
EXTRACTED H_2TCP . GOOD CURVES &
GOOD SEPARATIONS OBTAINED UNDER THE
SAME CONDITIONS.



THE BSA WAS THEN TRIED WITH
T & BIS ACIDS. THE T ACID IS
SILYLATED EASILY BUT THE BIS DID
NOT SILYLATE IN THF EVEN AFTER
HEATING AT 30°C FOR $\frac{1}{2}$ HOUR.
ALSO THERE WERE NO APPARENT PEAKS
FOR THE METHOXY DILYDROPHENOXY
ACETIC ACIDS.

IT
TRIED THIS PROCEDURE AGAIN LATER
AFTER MANY OTHER TYPES OF SAMPLES HAD
BEEN RUN THROUGH. TLB & DCP PEAKS
APPARENTLY HAVE GONE TOGETHER.

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022911

(Person NB # 7) 17

4/27/67

TDD LEVELS

ROW ON 720 WX @ 250° ON
IF -1

LOT	TDD
NI04-001	10 ppm
4-005	10
4-010	15
4-015	8
4-020	7
4-030	18
4-040	20
4-050	12

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032912

(Balderson NB # 8)

TDD IN N2TCP

1/31/69

2/4/69

OBJECTIVE: DETERMINE THE TDD
LEVEL IN N2TCP FROM THE PLANT
EQUIPMENT: FAM 720 W-2
FILAMENTS 2 METER AF-1-6500
ON GAS CHROM 2 RUN @ 270°C

PROCEDURE: WEIGH OUT 10 GRAMS
OF N2TCP INTO A 2 OZ BOTTLE.
ADD 10 ml CHCl_3 AND 2-3
mls. H_2O . ACIDIFY SLOWLY AND
WITH SHAKING TO A pH OF 1-2.
SHAKE FOR 5-10 MINUTES.

ADD 5 ml 2N (OR 25%) NaOH
AND AGAIN SHAKE FOR 5-10 MIN.
LET STAND UNTIL FULLY SEPARATED.

WITHDRAW THE BOTTOM CHCl_3 LAYER
AND PLACE IN A 4 DRAM VIAL
EVAPORATE DOWN TO 2 MILLILITERS.
WITHDRAW 40 μl AND INJECT
MEASURE PEAK HEIGHT AND CALCULATE
PPM TDD.

3 RUNS
STD. PK HTS. 57 mm, 60 mm, + 60 mm
B# 149 NIL
B# 154 NIL

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M. Wilson
2-21-69

C22913

27)

(Balderson NS #10)

TOD STANDARD:

3/2/70 OBJECTIVE: PREPARE A TOD STANDARD
IN CHLOROFORM FROM PURE SOLID
SUPPLIED BY DAN THROUGH AG
RESEARCH.

6.60 mg

6.26 mg

0.34 mg DILUTED TO 25 ml

0.0136 mg/ml

OR 13.6 μ g/ml

STD APPEARS CONTAMINATED

7.17

6.35

82 mg DIL TO 25 ml

0.0328 mg/ml

7.38

6.96

0.42 mg dil TO 25 ml

0.0168 mg/ml

Nath Wilson

5470

C22914

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TOD ANALYSES

WARD

3/6/70

LID

EXTRACTION DONE BY SHAKING 10 GRAMS
T WITH 20 ml CHCl_3 FOR 1 HOUR.
THE SAMPLE IS THEN CENTRIFUGED
AND THE CHLOROFORM WITHDRAWN. 25
ml 1 N NaOH ADDED AND THE
SAMPLE IS SHAKEN FOR 15 MINUTES.
CENTRIFUGED AND THE BOTTOM CHCl_3
LAYER WITHDRAWN NOTING VOLUME.
THE CHCl_3 LAYER IS EVAPORATED
TO DRYNESS AND 5% OF THE
AMOUNT OF CHCl_3 LAYER ADDED BACK
RUN ON 2 FT OV-1 ON GC @
190° FLAME. 1 μl X 20. ALL STEPS
RUN IN GLASS NO PLASTIC SHAKING
DONE IN GLASS STOPPERED BOTTLES

GLASS INJECTION INSERT

15.16 BENZENE

NL07-024 12 ppm

1.6 ppm

NL07-028 1.7 ppm

1.5 ppm

NL07-031 1.3 ppm

NL08-003 1.4 ppm

1.7 ppm

NL08-004 1.8 ppm

NL08-006 1.4 ppm

1.6 ppm

NL08-012 1.0 ppm

NL08-018 3.0 ppm

NL07-018 2.7 ppm

NL07-020 1.6 ppm

Marty Wilson
5470

022915

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29]

(Balderson NB #10)

NL08-020	2.7-2.5 ppm	
NL08-023	2.8 ppm	3.8% BIS
NL08-007	2.7 ppm	
NL08-026	1.0 ppm	
NL07-009	0.8 ✓	
NL07-011	0.9 ✓	
NL08-019	1.4	
NL08-025	2.2	
NL07-019	1.5	3.3% BIS
NL07-012	0.7-0.9-0.7 ✓	
NL06-026	1.2	
NL06-030	1.1	
NL06-012	1.6	
NL06-011	2.1	4.3% BIS
NL06-003	1.6	
NL06-007	1.8	
NL06-022	2.2	
NL-05-010	0.8	
NL-04-013	1.6	
NL-07-013 ✓	1.1	3.8% BIS
NL-06-020	0.8	
NL-04-019	1.4	
NL-05-005	1.8	
NL-08-023	2.9	
NL-08-027	1.6	
NL-08-007	2.0	
NL-07-013 ✓	0.9 ✓	3.8% BIS

W. A. Wilson 3470

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SUBJECT TO PROTECTIVE ORDER

100014

(Balderson NB #10) 13d

7% Bis

NL07-023 0.7 ppm

NL07-014 9.7 ppm

NL07-015 APPROX 22.0 ppm

NL08-028 1.7

~~NL06-020 0.4 ppm~~

{ NL06-015 ✓ 1.1 ppm }

{ NL06-015! 1.4 ppm }

NL07-021 1.5 ppm

NL08-021 1.4 ppm

NL06-013 0.9 ppm

NL06-028 0.3 ppm

NL05-009 1.0 ppm

NL06-023 0.7 ppm

NL05-032 0.5 ppm

NL08-033 1.3 ppm

NL08-030 1.1 ppm

NL08-031 1.3 ppm

NL06-038 0.5 ppm

NL08-029 1.1 ppm

NL03-007 4.4 ppm

NL04-024 0.6 ppm

NL05-013 1.0 ppm

NL05-014 0.7 ppm

NL05-036 0.8 ppm

NL06-006 0.5 ppm

NL06-034 0.9 ppm

NL06-020 0.8 ppm

315

3.9% Bis-T

5

70

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000017

Marty Wilson
5-4-70

311

(Balderson NB # 10)

NL06-024	0.8 ppm
NL07-024-029 (CURT)	1.2 ppm
NL05-003	0.9 ppm
DOW	0.5 ppm
NL01-005	10 ppm
NL01-006	9.0 ppm
NL08-022	2.6 ppm
mm-120639	0.5 ppm
NL07-010	1.1 ppm
NL08-034	3.8 ppm
mm-120619	0.6 ppm
NL07-030	2.8 ppm
NL06-014	2.1 ppm
NL08-005	2.4 ppm

Marty Wilson
5470

C22918

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TDD

10/30/70

INSTRUMENT 5754A FLAME.

2 FT 3% OV-1 ON

ABW - DMCS CHROMOSORB G

COLUMNS. COLUMN TEMPERATURE

200°C, INT 260°C, FLAME DETECTOR

265°C. AIR FLOW ROTAMETER

3.5 - 4.0, HELIUM ROTAMETER

2.9 TOP OF BALL, HYDROGEN

FOR MAXIMUM SENSITIVITY

@ 2.4 ROTAMETER (TOP OF BALL)

RANGE 10 ATTEN X2

PEAK HT. 5 μ / OF 16.8 μ /

TDD IN CHCl₃ GIVES PK

HT OF ~ 25 CHART DIVISION

(2.5 INCHES)

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022919

11/4/70

TDD CONTAMINATION LEVEL IN BLDG

OBJECTIVE: DETERMINE AS WELL AS POSSIBLE THE TDD LEVEL ON VARIOUS PIECES OF EQUIPMENT & IN THE BUILDING ITSELF. IN SLOG 92. PROCEDURE USED WAS TO MARK OFF AN AREA GENERALLY 1 SQ FOOT IN AREA AND WIPE WITH BENZENE SOAKED "KIM-WIPES" 10 TIMES. THE "KIM WIPES" THEN EXTRACTED WITH ϕ IN THE LAB TO RUN FOR TDD LEVELS. THE BENZENE EXTRACTED WAS WASHED WITH DILUTE NaOH , AND SEPARATED & FILTERED. BENZENE EVAPORATED OFF

#1 EMILCO DOOR 40 $\mu\text{g}/\text{sq ft}$ ^{TOO MUCH} MAX (NATL TONS 1)
 #2 2ND LEVEL GRATING 0.5 $\mu\text{g}/\text{sq ft}$ ^{1.5} MAX (NO DETECT)
 #3 TCP: W/T: EXT 14 $\mu\text{g}/\text{sq ft}$ MAX (LOWER TO NO DE)
 #4 ACIDIFIER INT 13 $\mu\text{g}/\text{sq ft}$ MAX (9/13)
 #5 3rd LEVEL STEEL (VENT) 11 $\mu\text{g}/\text{sq ft}$ MAX
 #6 PAN FILTER (2 LEAVES) NO TRACE
 #7 PAN FILTER HOPPER (DIRTY WITH T) 34 $\mu\text{g}/\text{sq ft}$
 #8 AUTOCLAVE HORIZ: TC NOT DETECTABLE
 #9 STILL VERT SIDE NONE DETECTABLE
 #10 1st LEVEL VERT STEEL TOO MUCH CONTAMINATION

C22920

CAN NOT TELL WHAT IS PRES

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6)

(Balderson NB 70-1T)

TDD FROM PLANT.

n/robo

THESE SAMPLES TAKEN AFTER
CLEANING IS ESSENTIALLY COMPLETE.
THESE WILL BE RUN BY A
MODIFIED PROCEDURE TO REMOVE
MOST OF THE IMPURITIES AWAY
FROM THE TDD. THE KIM-WIPES
ARE PLACED IN A FLAT
BOTTOM FLASK. 20 ml IN H_2SO_4
AND 70 ml H_2O ADDED. AN
AIR CONDENSER IS ATTACHED
AND THE SAMPLE HEATED TO
REFLUX FOR $\sim 2\frac{1}{2}$ HOURS. 20
ml TOULENE ADDED & REFLUXED
FOR ANOTHER HOUR. THE MIXTURE
IS THEN TRANSFERRED HOT TO
A SEP FUNNEL AND THE WATER
LAYER DRAWN OFF & DISCARDED.
THE TOULENE LAYER IS THEN
WASHED TWICE WITH 25 ml
 H_2O & IS WITH 10 ml CONC H_2SO_4
THE TOULENE IS THEN EVAP
TO DRYNESS. SAMPLE DISSOLVED
IN 10 ml PET ETHER (HEAT) &
TRANSFERRED TO A VIAL. EVAP
DOWN TO 1 ml & RUN

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C22921

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(Balderson NB 70-1T) 17

SAMPLE No	POINT	TOD
1	BRICK FLOOR GROUND LEVEL	NIL
2	PAW VERT. SIDE 2 nd EXTR.	NIL
3	EMICO FILTER DOOR	NIL
4	HORIZ TOP DRIER SCREW FEED	NIL
5	PAW VERTIKL SIDE 1 st EXTR	0.1-0.2 μ g
6	TOP #1 REACTOR	NIL
7	SIDE OF ELEVATOR REWORK CHUTE	NIL
8	GRATING 3 rd LEVEL	2.5 μ g/54 ft
9	VERT STEEL EXTR FEED TANK	0.7 μ g
10	VERT STEEL EXTR FILTER FD TK	< 1.0 μ g

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C22922

2-24-69 TOD Analysis
2-25-69

Object: To analyze 7 samples for percent TOD. Samples had been analyzed in St. Louis and TOD reported in the following samples:

#	TOD	%	Total Phenols	
149	4.3		20.9	
150	14.		21.5	A
151	11.5		20.64	B
152	3.1		22.3	B
154	2.6		30.7	A
155	2.3		30.5	A
157	6.5		26.2	B

Procedure:

- (1) Five gram sample was weighed into small vial. H_2O added until sample was dissolved (all samples were liquid) One ml of C_6H_6 was added and mixture was shaken for 10 min. The same sample was duplicated except one ml of $CHCl_3$ was used.
- (2) Step one was repeated with a one hour hold in automatic stirrer.
- (3) Step one repeated with heating of sample.

F. Bellerson

022923

3/11/69

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Note - this may be 1968 product ?

due to early run date in 69.

Bill Udert & Probable Run St. Louis. Samples

279
4/12/69

Instrument Settings:

F+M 720
 Column Temp 250°C
 Inj. Temp 250°C
 Det. Temp. 300°C
 Flow 75 ml/min
 Vacuum on exit port
 175 m²
 Att. 1X

Sample	Solvent	SHAKE Time	TDD
# 148	C ₆ H ₆	10 min	N.I.
# 148	CHCl ₃	10 min.	N.I.
# 149	C ₆ H ₆	10 min	N.I.
# 149	CHCl ₃	10 min	N.I.
# 151	C ₆ H ₆	1 Hour	N.I.
# 151	CHCl ₃	1 Hour	N.I.
# 151	C ₆ H ₆	10 min	N.I.
# 151	CHCl ₃	20 min	N.I.

Standard solution produced a TDD peak.

Possible problem in extraction

Marty's Method
 May be No Good?

P. Balaban
 3/11/69

022924

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NOT TO BE DISSEMINATED OUTSIDE THE FBI

3-18-19-69

TDD Analysis

Instrument Conditions

5750 A
Flame
265°C column
Flame Det. 295°C
Dry Port 310°C
X1 10²

Setting

Air #4 B#
H₂ A#3½ B#
Helium A#2½ B#
OV-17 column

Procedure #1

- (1) 5 gram sample; 3-4 ml H₂O
shake, add 1 ml CCl₄
Shake 10 min.
- (2) Inject Sol from bottom layer

Sample

293

240

239

248

296

300

303

Std peak height = 144

p.p.m.

At 0.5 ppm

At 0.6 ppm

0.6 ppm

0.5 ppm

0.3 ppm

0.4 ppm

0.5 ppm

F. B. Wilson
4/21/69

Procedure #II

Take a 10 gram sample, add water to dissolve then add 5 ml CCl_4 and shake 15 - 20 min. Put CCl_4 layer in small vial via syringe, then evaporate to 1 ml. Do not go to dryness and add back. Inject 5 μl sample into Chromatograph. Measure peak height and calculate ppm.

3-21-69

Instrument Conditions the same as on page 6.

<u>Sample</u>	<u>TDD</u>
#1	0.4 ppm
264	0.6 ppm
281	0.4 ppm
283	0.4 ppm
264	0.8 ppm
247	1.1 ppm

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022926

4-16-69

TDD Analysis

Instrument conditions: Refer to page 6 and procedure Book #2

Std peak height = 151.5 mm. Std = 1/2
Calculations:

$$\text{ppm TDD} = \frac{(\text{peak height of unknown}) \left(\frac{151.5}{10000} \right) (1 \text{ ml})}{(\text{peak height of std.}) \left(\frac{151.5}{10000} \right) (1 \text{ ml})}$$

Sample	PH height	ppm TDD	(3) Sample
341	3.5	0.2	
276	1	0.1	
342	2.75	0.2	
272	3.0	0.2	
394	3	0.2	
252	1.75	0.1	
261	1.75	0.1	
395	3.5	0.2	
396	3	0.2	
242	3.15	0.3	
250	4	0.3	

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022927

Analysis of Solution TSP for TDD

Objective : To analyze TSP for % TDD
to confirm low level TDD. Question
was raised when control lot reported
8 ppm TDD in Tacid.

Procedure

- ① Five gram sample in small vial;
add H₂O (several ml) to dissolve sample,
shake well.
- ② Add 1 ml CHCl₃ and shake for 10 mins.
Let separate and inject 5ul from CH
layer.
- ③ Standard solution - 5ul size sample from

<u>Sample</u>	<u>TDD in ppm</u>
477	0.6
479	ND
489	0.2
440	0.5
443	0.5
510	0.5
517	0.2

R. Delepper
6/13/67

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C22928